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(54) Title: PESTICIDAL FORMULATIONS

(57) Abstract

Pesticidal formulations comprising a photosensitive insecticide or a photosensitive herbicide or photosensitive insecticide or herbicide synergist encapsulated in yeast, and their use in the control of insect and acarine pests and rodents are described.

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# PESTICIDAL FORMULATIONS

The present invention relates to formulations for stabilising light sensitive substances, particularly pesticides and/or pesticides and herbicide synergists, and the use of such formulations in the control of pests.

European patent No. 85805B discloses encapsulated products involving a microbial capsule, and specifically a yeast capsule, and a method for producing such encapsulated products. The encapsulated products are formed by treating the grown microbe with an organic lipid-extending substance in which the material being encapsulated is soluble, and simultaneously and/or subsequently contacting the microbe with the material to be encapsulated. It is mentioned that the encapsulation method is particularly suitable for producing encapsulated dyes but other examples of material to be encapsulated include adhesives, adhesive components, pharmaceuticals, flavours, rodenticides, insecticides, herbicides, fungicides and odiferous materials.

European patent specification No. 242135A discloses a modification of this microbial encapsulation technique in which the grown intact microbe is treated with an encapsulatable material in liquid form whereby the encapsulatable material diffuses into the microbial cell without causing total lysis thereof. The process is carried out in the absence of a lipid-extending substance (as defined in European patent No. 85805B). The products of this encapsulation technique are described as being usually more stable than non encapsulated materials and to give products with controlled release characteristics advantageous for the administration of drugs, pheromones and pesticides. However, it was not known that one could decrease the sensitivity of a substance to light (ie. its photosensitivity) by simply encapsulating the substance without incorporating further materials such as absorbers or light scatterers.

It has now been found that light sensitive/photosensitive substances, such as the synergist piperonyl butoxide and insecticides extracted from pyrethrum (the pyrethrins) when encapsulated in yeast become significantly less light sensitive and can be used for applications not previously available to them.

In the present context a photosensitive substance is a substance which degrades rapidly on exposure to daylight and would typically have a half-life of less than 5 hours when this is measured under test conditions, and particularly is a substance which has a half-life of less than 1 hour and more particularly is a substance which has a half-life of less than 40 minutes. Typically the use of the photosensitive substance is limited by its photosensitivity. Suitable test conditions are provided by applying the substance to an inert surface (such as glass) and exposing the deposit to controlled artificial light conditions. A suitable source of artificial sunlight is provided, by the use of a "Hanau Suntester" (Heraeus Suntest Lamp, accelerated exposure table unit), as described hereinafter.

Accordingly, the present invention provides a formulation which comprises a photosensitive active ingredient encapsulated in yeast. Suitably, the photosensitive active ingredient is a photosensitive insecticide or a photosensitive herbicide or an insecticide synergist or herbicide synergist encapsulated in yeast. Suitably the photosensitive insecticide has a half-life of less than 40 minutes and is a member of the pyrethrin class of insecticides. Further insecticides which may with advantage be included in the formulation of the present invention are those which have a half-life of greater than 1 hour and include pyrethroids such as the allethrins (such as bioallethrin, S-bioallethrin and esbiothrin), the resmethrins (resmethrin, bioresmethrin), lipid amides (for example as described in European Patent Specifications 0369762, 111105, 164187, 194764, 209289, 225011, 251472, 269457 and 317188) and pyrethroids which have some degree of light stability, for example, permethrin, α-cypermethrin and deltamethrin.

A suitable photosensitive herbicide is trifluralin. A suitable rodenticide is cholecalciferol (vitamin $D_3$). Suitably the insecticide or herbicide synergist is piperonyl butoxide (5-[2-(2-butoxyethoxy)-ethoxymethyl]-6-propyl-1,3-benzodioxole). Preferably the encapsulated material is piperonyl butoxide or a mixture of this with a pyrethrum extract or a member of the allethrin class of insecticides. Preferably, the herbicide, insecticide or herbicide or insecticide synergist is encapsulated in yeast by the method of European patent specification No. 242135A or European patent No. 85805B.

The precise amount of encapsulated material to be applied to a locus would depend on the inherent activity of the material against the insect or weed species to be controlled, the method of application and the nature of the formulation to be applied and whether or not piperonyl butoxide is included in the formulation, but in general the following quantities would be applied : pyrethrins between 50 and 300 grams per hectare (two and twelve ounces per acre), piperonyl butoxide between 50 and 1200 grams per hectare (0.125 and 3lbs per acre) and lipid amides between 5 and 150 grams per hectare (0.0125 and 0.4lbs per acre).

Formulations of the present invention may be applied as solid formulations in a mixture with a powdered solid inert carrier, for example suitable clays, kaolin, bentonite, attapulgite, absorbant carbon black, talc, mica, chalk, gypsum, tricalcium phosphate, powdered cork, magnesium silicate, vegetable carriers, starch and diatomaceous earths.

Wettable powders comprise an inert solid carrier(s), optionally one or more surface active agents, and/or stabilisers and/or anti-oxidants.

Wettable powders will normally contain from 5 to 95% by weight of the active ingredient, and are diluted, for example with water, before use.

Aqueous suspensions of a formulation of the present invention may comprise a suspension in water together with suspending, stabilising or other agents. The suspensions or solution may be applied per se or in a diluted form in known fashion.

Aerosol sprays may be prepared as a suspension of the active ingredient in water in the presence of an aerosol propellant, such as carbon dioxide. Pour-on formulations may be made as a suspension of a formulation of the present invention in water.

Baits may comprise a formulation of the present invention, optionally admixed with additional edible material in a suitable binding matrix.

The formulations of the present invention may be used to control pests such as arthropod (insect and acarine) pests, and rodents.

Thus the present invention provides a method for the control of arthropod or rodent pests which comprises administering to the arthropod or rodent pest or to its environment a pesticidally effective amount of a pesticidal formulation of the present invention. The present invention also provides a method for the control and/or eradication of arthropod infestations of animals and/or of plants, (including trees), and/or stored products which comprises administering to the animal or locus an effective amount of an arthropodicidal formulation of the present invention. The present invention further provides pesticidal formulations of the present invention for use in controlling pests in human and veterinary medicine, in public health control and in agriculture.

The formulations of the present invention may also be used to control weeds.

Thus, the present invention provides a method for the control of weeds which comprises administering to the weed or to its environment a

herbicidally effective amount of a herbicidal formulation of the present invention.

The insecticidal formulations of the present invention are of particular value in the protection of field, forage, plantation, glasshouse, orchard and vineyard crops, of ornamentals and of plantation and forest trees, for example, cereals (such as maize, wheat, rice, sorghum), cotton, tobacco, vegetables and salads (such as beans, cole crops, curcubits, lettuce, onions, tomatoes and peppers), field crops (such as potato, sugar beet, ground nuts, soyabean, oil seed rape), sugar cane, grassland and forage (such as maize, sorghum, lucerne), plantations (such as of tea, coffee, cocoa, banana, oil palm, coconut, rubber, spices) orchards and groves (such as of stone and pip fruit, citrus, kiwifruit, avocado, mango, olives and walnuts), vineyards, ornamental plants, flowers and shrubs under glass and in gardens and parks, forest trees (both deciduous and evergreen) in forests, plantations and nurseries.

They are also valuable in the protection of timber (standing, felled, converted, stored or structural) from attack by sawflies (e.g. Urocerus) or beetles (e.g. scolytids, platypodids, lyctids, bostrychids, cerambycides, anobiids).

They have applications in the protection of stored products such as grains, fruits, nuts, spices and tobacco, whether whole, milled or compounded into products, from moth, beetle and mite attack. Also protected are stored animal products such as skins, hair, wool and feathers in natural or converted form (e.g. as carpets or textiles) from moth and beetle attack; also stored meat and fish from beetle, mite and fly attack.

The insecticidal compositions of the present invention are thus useful in the control of arthropods, e.g. insects and acarines in any environment where these constitute pests, e.g. in agriculture, in animal husbandry, in public health control and in domestic situations.

Insect pests include members of the orders Coleoptera (e.g. Anobium, Ceutorhynchus, Rhynchophorus, Cosmopolites, Lissorhoptrus, Meligethes, Hypothenemus, Hylesinus, Acalymma, Lema, Psylliodes, Leptinotarsa, Gonocephalum, Agriotes, Dermolepida, Heteronychus, Phaedon, Tribolium, Sitophilus, Diabrotica, Anthonomus or Anthrenus spp.), Lepidoptera (e.g. Ephestia, Mamestra, Earias, Pectinophora, Ostrinia, Trichoplusia, Pieris, Laphygma, Agrotis, Amathes, Wiseana, Tryporysa, Diatrae, Sporganothis, Cydia, Archips, Plutella, Chilo, Heliothis, Spodoptera or Tineola spp.), Diptera (e.g. Musca, Aedes, Anopheles, Culex, Glossina, Simulium, Stomoxys, Haematobia, Tabanus, Hydrotaea, Lucilia, Chrysomia, Callitroga, Dermatobia, Gasterophilus, Hypoderma, Hylemyia, Atherigona, Chlorops, Phytomyza, Ceratitis, Liriomyza and Melophagus spp.), Phthiraptera (Malophaga e.g. Damalina spp. and Anoplura e.g. Linognathus and Haematopinus spp.), Hemiptera (e.g. Aphis, Bemisia, Phorodon, Aeneolamia, Empoasca, Parkinsiella, Pyrilla, Aonidiella, Coccus, Pseudococus, Helopeltis, Lygus, Dysdercus, Oxycarenus, Nezara, Aleurodes, Triatoma, Psylla, Mysus, Megoura, Phylloxera, Adelyes, Niloparvata, Nephrotetix or Cimex spp.), Orthoptera (e.g. Locusta, Gryllus, Schistocerca or Acheta spp.), Dictyoptera (e.g. Blattella, Periplaneta or Blatta spp.) Hymenoptera (e.g. Athalia, Cephus, Atta, Solenopsis or Monomorium spp.), Isoptera (e.g. Odontotermes and Reticulitermes spp.), Siphonaptera (e.g. Ctencephalides or Pulex spp.), Thysanura (e.g. Lepisma spp.), Dermaptera (e.g. Forficula spp.), Pscoptera (e.g. Peripsocus spp.) and Thysanoptera (e.g. Thrips tabaci),. Acarine pests include ticks, e.g. members of the genera Boophilus, Ornithodorus, Rhipicephalus, Amblyomma, Hyalomma, Ixodes, Haemaphysalis, Dermacentor and Anocentor, and mites and manges such as Acarus, Tetranychus, Psoroptes, Notoednes, Sarcoptes, Psorergates, Chorioptes, Eutrombicula, Demodex, Panonychus, Bryobia, Eriophyes, Blaniulus, Polyphagotarsonemus, Scutigerella, and Oniscus spp.

The formulations of the present invention can be used in the control of larvae, for example, in the form of a bait containing a suitable larvicide such as permethrin.

The formulations of the present invention which contain a rodenticide can be used in the control of rodents.

The present invention will now be described in more detail by way of the following examples:

Examples 1-7 are formulations according to the present invention in which the method of yeast encapsulation followed is as given in the method of European patent specification 242135 in that the material to be encapsulated is mixed with the yeast in a liquid medium. Example 8 shows the results of tests to determine the light-stability of piperonyl butoxide encapsulated in yeast cells. Example 9 shows the results of biological tests on cockroaches.

Example 1

Spray dried formulation of yeast encapsulated Pyrethrum/Piperonyl butoxide

| | approx. % w/w |
|---|---|
| Pyrethrum Pale 25% | 10.8 |
| Piperonyl butoxide (PBO) | 29.3 |
| Bakers yeast | 56.0 |
| Synthetic silica (eg Sipernat 22S) | 3.9 |

This formulation is made from the following concentrate.

Concentrate:

| | approx. % w/w |
|---|---|
| Pyrethrum Pale 25% | 4.6 |
| PBO | 11.5 |
| Bakers yeast | 30.1 |
| Water | 53.5 |
| Preservative | 0.3 |

The formulation is made by mixing the concentrate with fumed silica (eg. Cab-o-sil M5) and water in the following ratio after which the formulation is passed through a spray dryer.

| | approx. % w/w |
|---|---|
| Concentrate | 24.8 |
| Cab-o-sil M5 | 1.0 |
| Water | 74.2 |

Example 2

Spray dried formulation of yeast encapsulated Pyrethrum/Piperonyl butoxide

The formulation is made by mixing the following concentrate with fumed silica (eg. Cab-o-sil M5) and water in the ratio given for Example 1.

Concentrate:

| | approx. % w/w |
|---|---|
| Pyrethrum Pale 25% | 10.8 |
| PBO | 29.3 |
| Bakers yeast | 52.8 |
| Silica (eg Cab-o-sil M5) | 7.0 |
| Preservative | 0.1 |

Examples 1 and 2, are of general applicability for additions of up to 0.7g active ingredient per g of yeast.

Example 3

Permethrin Powder (Wettable)

| | approx. % w/w |
|---|---|
| Permethrin | 11.9 |
| C9 primary alcohol | 7.9 |
| Bakers yeast | 80.1 |
| Preservative (formalin) | 0.1 |

Made from a washed and spray dried Permethrin concentrate (No anti caking agent required) as follows:

| | approx. % w/w |
|---|---|
| Permethrin | 15.3 |
| C9 primary alcohol | 10.2 |
| Bakers yeast | 38.5 |
| Water | 35.7 |
| Formalin | 0.3 |

Example 4

Dust (made from powder concentrate, Example 3)

| | final approx % w/w |
|---|---|
| Permethrin tech. | 0.5 |
| Solvent (C9 alcohol, Aromasol H) | 0.2 |
| Bakers yeast | 1.4 |
| Silica (Cab-o-sil M5) | 1.8 |
| Talc | 96.1 |

Example 5

Aqueous suspensions

Various dilutions may be required, depending on final concentration. Yeast tends to settle and hence a suspending agent may be used.

| (10% dilution) | approx. % w/w |
|---|---|
| Yeast concentrate: (14% yeast encapsulated permethrin, see Example 6) | 10.0 |
| Suspending agent eg. biopoly-saccharide, xanthan gum, Keltrol) | 0.3 |
| Water | 89.6 |
| Preservative (eg Bronidox L, 5-bromo-5-nitro, 1,3 dioxane in propylene glycol, Proxel GXL (aqueous diisopropylene glycol solution of approximately 20% 1,2-benzisothiazolin-3-one (B.I.T.) as sodium salt, or formalin. | 0.1 |

Levels of suspending agent will vary depending on the type used. This dilution may then be used as produced or as part of another formulation eg aerosol.

Example 6

Aerosols

| | approx. % w/w |
|---|---|
| i) Yeast concentrate (eg 14% Permethrin, see below) | 9.8 |
| Wetting agent (eg silicone wetter Tegoplant WT25) | 0.1 |
| Water | 88.0 |
| $CO_2$ | 2.1 |
| ii) Yeast concentrate (eg. 14% yeast encapsulated Permethrin, see below) | 60.0 |
| Butane 40 | 40.0 |

Yeast concentrate (14% yeast encapsulated Permethrin)

| | approx. % w/w |
|---|---|
| Permethrin | 14.0 |
| Aromatic solvent (Aromasol H) | 9.4 |
| Bakers yeast | 35.2 |
| Water | 41.1 |
| Preservative (formalin, Proxel GXL or Bronidox L) | 0.3 |

Example 7

Baits

| | approx. % w/w |
|---|---|
| i) Magnesium - Aluminium Silicate (Low grade mica) eg Vermiculite | 58.6 |
| Syrup | 40.0 |
| Yeast concentrate (as below) | 1.4 |

Concentrate:

| | approx. % w/w |
|---|---|
| Permethrin | 7.2 |
| Aromasol H | 7.2 |
| Bakers yeast | 28.5 |
| Water | 57.0 |
| Antifoam (eg. Silcolapse 5000) | 0.1 |

ii) Alternative base matrix

| | approx. % w/w |
|---|---|
| Soya flour | 20.0 |
| Fish meal | 20.0 |
| Sugar | 20.0 |
| Paraffin wax | 11.0 |
| Soya oil | 20.0 |
| Yeast concentrate | 9.0 |

Larvicides - may be used as a diluted concentrated sample ie up to 40% active ingredient/solvent mix, or as a low encapsulated level eg 1 - 2% (Permethrin) or lower with other active ingredients.

Example 8

Experiments

Determination of light-stability of Piperonyl Butoxide encapsulated in yeast cells

A. Stability on glazed tiles

Tests were carried out to determine the light stability of piperonyl butoxide encapsulated in yeast cells. The following method was also applied to piperonyl butoxide/pyrethrins mixtures as well as to the more light-stable pyrethroids.

Method

A dilution in water was prepared, to give a piperonyl butoxide content of 6mg/ml (or 20mg/ml in piperonyl butoxide/pyrethrins formulations). A 6cm circle was marked out onto a 10.5 x 10.5cm glazed white ceramic tile and 0.5ml of the well mixed suspension applied by pipette evenly within the marked circle. The tile was left overnight on a level surface to dry.

The tile was then placed in a Hereaus Suntest Lamp ("Hanau Suntester"), water-cooled to give a maximum temperature of 30°C and exposed for an exact time which was then recorded. The light on the tile surface was 60,000 lux, 2.5 $mW/cm^2$ UVA. The tile was taken from the lamp and the deposit removed with small swab of cotton wool soaked in methanol. These were transferred to a 50ml stoppered conical flask and the methanol evaporated in a current of air. 10ml of methanol was pipetted onto the deposit and placed in a sonic bath for 5 minutes. The resultant solution was filtered and subjected to GLC analysis comparing chromatograms with a 0.02 percent solution of piperonyl butoxide.

A control was carried out by applying 0.5ml of a 0.6 percent solution of technical piperonyl butoxide within the 6cm circle and carrying out the test as outlined.

The Pyrethrins/PBO sample used was as follows:

| Sample PYR1 | % w/w |
|---|---|
| Pyrethrum 25% pale | 5.5 (10% of total unencapsulated) |
| PBO | 13.5 (16% of total unencapsulated) |
| Bakers yeast | 35.6 |
| Water | 45.4 |

The permethrin/PBO sample used was as follows:

| Sample PRM2 | % w/w |
|---|---|
| Permethrin | 8.0 (7.8% internal) |
| PBO | 8.0 (7.4% internal) |
| Yeast | 28.5 |
| Water | 55.5 |

Results

Results from the UV stability testing of Pyrethrins/PBO and

TABLE 1

(YEAST ENCAPSULATED) PYRETHRINS/PIPERONYL BUTOXIDE
STABILITY ON GLAZED TILES EXPOSED TO HANAU SUNTESTER

Nominal Application on tiles 1.05 mg PYRETHRINS ) PYR1
10.2 mg PBO )

| Minutes of exposure | 5 mins | 10 mins | 15 mins | 20 mins | 30 mins |
|---|---|---|---|---|---|
| PYRETHRINS mg/tile | 0.63 | 0.38 | 0.20 | 0.16 | 0.11 |
| PBO mg/tile | 9.7 | 10.3 | 10.3 | 9.5 | 9.7 |

TABLE 2

(YEAST ENCAPSULATED) PYRETHRINS/PIPERONYL BUTOXIDE
STABILITY ON GLAZED TILES EXPOSED TO HANAU SUNTESTER

Nominal Application on tiles — 1.05 mg PYRETHRIN, 10.2 mg PBO ) PYR1
Control — 10.7 mg PBO

| Exposure Time | 0.5 hours | 1.0 hours | 2.0 hours | 3.0 hours |
|---|---|---|---|---|
| Sample PYR1 | | | | |
| mg PBO | - | 9.4 | 8.3 | 7.8 |
| Control | | | | |
| mg PBO | 5.2 | NT | NT | NT |

NT = not tested

## TABLE 3

(YEAST ENCAPSULATED) PERMETHRIN/PIPERONYL BUTOXIDE
STABILITY ON GLAZED TILES EXPOSED TO HANAU SUNTESTER

Nominal Application on Tiles — 3.0 mg PERMETHRIN, 3.2 mg PIPERONYL BUTOXIDE ) PRM2
Control — 3.0 mg PERMETHRIN
Control — 10.7 mg PIPERONYL BUTOXIDE

| Exposure Time | 0.5 hours | 1 hour | 2 hours | 3 hours | 7 hours |
|---|---|---|---|---|---|
| Sample PRM 2 | | | | | |
| PRM mg/tile | NT | 2.9 | 2.9 | 2.7 | 2.2 |
| PBO mg/tile | NT | 3.0 | 2.9 | 2.6 | 2.3 |
| Control | | | | | |
| PRM mg/tile | 2.7 | 2.4 | NT | NT | NT |
| Control | | | | | |
| PBO mg/tile | 5.2 | | | | |

NT = not tested

B. Stability on glass

Testing was carried out on Piperonyl Butoxide encapsulated in yeast to determine the overall stability profile on glass. Experiment A above indicated the half life of PB to be increased when encapsulated in yeast and applied to glazed tiles, but no value was determined. This experiment was designed to obtain a half-life (T1/2 value) for the formulation used.

Sample: Piperonyl Butoxide (22.1% w/w internal, 0.7% w/w external) in bakers yeast.

Method

The formulation was diluted with water and applied to glass plates at a rate of 50mg/plate. The dilution was applied weight wise as small droplets over the plate surface and allowed to dry before being placed under the Suntest lamp for intervals up to 36 hours. The total levels of PB remaining were then determined using the method outlined in A.

TABLE 4

Application rate 0.14mg/6cm plate = 50mg $m^2$

| Time (hours) | % of active remaining after exposure |
|---|---|
| initial | 100 |
| 2 | 97 |
| 5 | 84 |
| 8 | 76 |
| 12 | 69 |
| 16 | 66 |
| 36 | 52 |

Result

T 1/2 value for yeast encapsulated PBO of about 35h.

A typical value for a control would be about 30 minutes.

Example 9

Mortality testing of yeast encapsulated pyrethrum/piperonyl butoxide against cockroaches

Formulations similar to Examples 1 or 2 were prepared containing 4.5% technical pyrethrin and 13.5% piperonyl butoxide. Serial dilutions were prepared giving solutions containing 0.2-0.02% w/v pyrethrins. 0.5ml aliquots of these dilutions were applied to glazed tile surfaces.

Ten cockroaches (male, Blatella germanica) were exposed to wet deposits of these formulations for 30 seconds and the mortality recorded after 6 days compared to a pyrethrin/piperonyl butoxide formulation (Pybuthrin 5/50 EC.) which is an emulsifiable concentrate containing 5% pyrethrin and 50% piperonyl butoxide.

Results

TABLE 5

| Formulation | Pyrethrin w/v % | mean % 6 day Mortality |
|---|---|---|
| (Yeast Encapsulated pyrethrum/ piperonyl butoxide) | 0.2 | 100 |
| | 0.1 | 100 |
| | 0.05 | 100 |
| Pybuthrin 5/50 EC. | 0.2 | 100 |
| | 0.1 | 100 |
| | 0.05 | 100 |

CLAIMS

1. A formulation comprising a photosensitive active ingredient encapsulated in yeast.

2. A formulation comprising a photosensitive insecticide or a photosensitive herbicide or an insecticide synergist encapsulated in yeast.

3. A formulation according to claim 2 wherein the photosensitive insecticide is a member of the pyrethrin and/or light sensitive pyrethroid classes of insecticides.

4. A formulation according to claim 3 wherein the light sensitive pyrethroid is a member of the allethrin or resmethrin class of insecticides.

5. A formulation according to any of claims 1 to 4 which contains a lipid amide, permethrin or deltamethrin.

6. A formulation according to any preceding claim wherein the insecticide or herbicide synergist is piperonyl butoxide.

7. A formulation according to any preceding claim wherein the encapsulated material is piperonyl butoxide with pyrethrum extract or with a member of the allethrin class of insecticides.

8. A formulation according to any preceding claim wherein the photosensitive active ingredient is encapsulated in yeast by mixing the yeast with the photosensitive active ingredient in a liquid medium.

9. A formulation according to any preceding claim in the form of a spray dried formulation, a concentrate, a powder, a dust, an aqueous suspension, an aerosol or a bait.

10. A method of controlling pests by administering to the pest or its environment a pesticidally effective amount of a photosensitive pesticide encapsulated in yeast.

11. A method for controlling weeds comprising administering to the weed or to its environment a herbicidally effective amount of a photosensitive herbicide encapsulated in yeast.

# INTERNATIONAL SEARCH REPORT

International Applica ..No PCT/GB 91/00944

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl.5 A 01 N 25/28

## II. FIELDS SEARCHED

Minimum Documentation Searched[7]

| Classification System | Classification Symbols |
|---|---|
| Int.Cl.5 | A 01 N B 01 J |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

## III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

| Category * | Citation of Document,[11] with indication, where appropriate, of the relevant passages[12] | Relevant to Claim No.[13] |
|---|---|---|
| X | GB,A,2162147 (DUNLOP LTD) 29 January 1986, see page 2, lines 10-16,99-105; examples XII,XVI; claims | 1-5,8 |
| X | US,D, 498208 (J.L. SHANK et al.) 13 April 1976, see column 3, lines 18-40; claims | 1,2,8 |
| A | EP,A,0085805 (DUNLOP LTD) 17 August 1983 (cited in the application) | |
| A | EP,A,0242135 (AD2 LTD) 21 October 1987 (cited in the application) | |

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## IV. CERTIFICATION

| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report |
|---|---|
| 12-09-1991 | 10. 10. 91 |
| International Searching Authority: EUROPEAN PATENT OFFICE | Signature of Authorized Officer: miss T. MORTENSEN |

Form PCT/ISA/210 (second sheet) (January 1985)

# ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. GB 9100944 SA 48428

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 26/09/91 The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
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| GB-A- 2162147 | 29-01-86 | None | |
| US-D- 498208 | | None | |
| EP-A- 0085805 | 17-08-83 | AU-A- 9072582<br>JP-A- 58107189 | 26-05-83<br>25-06-83 |
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82